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Nishizawa; Junichi, Sendai, Japan Inventor(s):

Suzuki; Rensaku, Sendai, Japan Aizawa; Kenji, Sendai, Japan

Applicant/Assignee Inquire Regarding

Shin Tohoku Chemical Industries

Inc., Japan

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422/169; 423/219; 423/351;

Original: 055/031; 055/033; 055/075; 055/208; 055/389; 422/169; 423/219;

423/351;

Field of Search: 055/68,75,208,387,389,31,33

209/567,576,577,580,581,589 252/455

Z 423/219,351 422/169

Dec. 29, 1978 JP1978000162338 **Priority Number**

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Genuine Article#: YP621 Number of References: 16 Title: Conserving toxic ammoniacal nitrogen in manure using natural zeolite tuff: A comparative study Author(s): Dwairi IM (REPRINT) Corporate Source: YARMOUK UNIV, DEPT EARTH & ENVIRONM SCI/IRBID//JORDAN/ (REPRINT) Journal: BULLETIN OF ENVIRONMENTAL CONTAMINATION AND TOXICOLOGY, 1998, V60 , N1 (JAN), P126-133 Publication date: 19980100 ISSN: 0007-4861 Publisher: SPRINGER VERLAG, 175 FIFTH AVE, NEW YORK, NY 10010 Document Type: ARTICLE Language: English Geographic Location: JORDAN Subfile: CC LIFE--Current Contents, Life Sciences; CC AGRI--Current Contents, Agriculture, Biology & Environmental Sciences Journal Subject Category: ENVIRONMENTAL SCIENCES; TOXICOLOGY Cited References: *AM PUBL HLTH ASS, 1971, STAND METH EX WAT WA *AM SOC AGR ENG, 1982, AGR ENG YB ANDREWS RD, 1993, P250, 4 INT C OCC PROP UT BOULDIN DR, 1981, 81 CORN U DEP AGR DWAIRI IM, 1992, V19, P7, DIRASAT U JORDAN B DWAIRI IM, 1997, IN PRESS ENV GEOLOGY DWAIRI IM, 1993, V8, P7, MUTAH J RES STUDIES DWIARI IM, 1987, THESIS U HULL ENGLAN FEBLES JA, 1991, ZEOLITES 91
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slow-release fertilizers. The use of NH//4** plus -phillipsite tuff offers

an option to the widely used soluble NH//4-fertilizers in agriculture to avoid environmental problems associated with nitrogen contamination of surface water and groundwater. (Author abstract) 20 Refs. Descriptors: Geochemistry; Volcanic rocks; Zeolites; Isotherms; Saturation (materials composition); Composition effects; Nitrogen fertilizers; Solubility; Ion exchange; Thermal effects Identifiers: Ammonium phillipsite tuff; Ammonium fertilizers; Exchange isotherms Classification Codes: 481.2 (Geochemistry); 482.2 (Minerals); 804.2 (Inorganic Components); 801.4 (Physical Chemistry) (Geology & Geophysics); 482 (Mineralogy & Petrology); 804 (Chemical Products); 801 (Chemical Analysis & Physical Chemistry) 48 (ENGINEERING GEOLOGY); 80 (CHEMICAL ENGINEERING) (Item 4 from file: 399) DIALOG(R) File 399:CA SEARCH(R) (c) 2001 AMERICAN CHEMICAL SOCIETY. All rts. reserv. 129067287 CA: 129(6)67287z **JOURNAL** Renewable, controlled and environmentally safe phosphorus release in soils from mixtures of NH4+-phillipsite tuff and phosphate rocks AUTHOR(S): Dwairi, I. M. LOCATION: Dep. Earth Environmental Sciences, Yarmouk Univ., Irbid, Jordan JOURNAL: Environ. Geol. (Berlin) DATE: 1998 VOLUME: 34 NUMBER: 4 PAGES: 293-296 CODEN: ENGOE9 ISSN: 1073-9106 LANGUAGE: English PUBLISHER: Springer-Verlag SECTION: CA219003 Fertilizers, Soils, and Plant Nutrition IDENTIFIERS: phosphorus ammonium phillipsite fertilizer soil, calcium phosphate fertilizer phosphorus ammonium phillipsite DESCRIPTORS: Zeolite group minerals... Ca2+ satd.; P release in soils from mixts. of NH4+-phillipsite tuff and phosphate rocks Fertilizer experiment... Phosphate rock... P release in soils from mixts. of NH4+-phillipsite tuff and phosphate CAS REGISTRY NUMBERS: 14798-03-9 biological studies, combined with phillipsite; P release in soils from mixts. of NH4+-phillipsite tuff and phosphate rocks 7440-23-5 biological studies, P and Na release in soils from mixts. of NH4+-phillipsite tuff and phosphate rocks 7723-14-0 biological studies, P release in soils from mixts. of NH4+-phillipsite tuff and phosphate rocks 12174-18-4 NH4+ contg.; P release in soils from mixts. of NH4+-phillipsite tuff and phosphate rocks 7757-93-9 P release in soils from mixts. of NH4+-phillipsite tuff and monocalcium phosphate (Item 5 from file: 8) DIALOG(R)File 8:Ei Compendex(R) (c) 2001 Engineering Info. Inc. All rts. reserv. E.I. No: EIP96103362007 04523164 Title: Removal of nutrients from sewage effluent in stabilization ponds

using natural zeolite

Author: Gharaibeh, S.H.; Dwairi, I.M. Corporate Source: Yarmouk Univ, Irbid, Jordan Source: Chemische Technik (Leipzig) v 48 n 4 Aug 1996. p 215-218 Publication Year: 1996 CODEN: CHTEAA ISSN: 0045-6519 Language: English Document Type: JA; (Journal Article) Treatment: X; (Experimental); A; (Applications) Journal Announcement: 9612W2 Abstract: Jordanian natural zeolitic tuff samples were tested as a tertiary treatment on the effluent of wastewater stabilization ponds using laboratory scale column methods. The results of the column method showed that the zeolitic tuff had high efficiencies for ammonium and phosphate removal, and low efficiencies for nitrate elimination. The highest removal value for ammonium was 78.6% using raw zeolitic tuff of 0.5 - 0.25 mm size fraction, while the highest removal for phosphate was 30.05% using Ca-form zeolitic tuff of 0.5 - 0.25 mm grain size. (Author abstract) 11 Refs. Descriptors: Sewage lagoons; Zeolites; Effluents; Wastewater treatment; Ammonium compounds; Phosphates; Nitrates; Particle size analysis; Effluents Identifiers: Natural zeolitic tuff; Stabilization ponds; Column methods; Ammonium removal; Phosphate removal; Nitrate elimination; Grain size Classification Codes: (Sewage Treatment); 452.3 (Industrial Wastes); 804.2 (Inorganic Components); 452.4 (Industrial Wastes Treatment); 943.3 (Special Purpose Instruments) (Sewage & Industrial Wastes Treatment); 804 (Chemical Products); (Mechanical & Miscellaneous Measuring Instruments) (POLLUTION & SANITARY ENGINEERING); 80 (CHEMICAL ENGINEERING); 94 (INSTRUMENTS & MEASUREMENT) (Item 6 from file: 399) DIALOG(R) File 399:CA SEARCH(R) (c) 2001 AMERICAN CHEMICAL SOCIETY. All rts. reserv. 120034882 CA: 120(4)34882a JOURNAL Jordanian zeolites: evaluation for possible industrial application of natural Aritain phillipsite tuffs AUTHOR(S): Dwairi, I. M. LOCATION: Yarmouk Univ., Jordan, JOURNAL: Dirasat - Univ. Jordan, Ser. B DATE: 1992 VOLUME: 19B NUMBER: 1 PAGES: 23-44 CODEN: DJSSE8 LANGUAGE: English SECTION: CA253005 Mineralogical and Geological Chemistry IDENTIFIERS: zeolitic tuff industrial use assessment Jordan, phillipsite rich tuff possible use Jordan DESCRIPTORS: Zeolite-group minerals... in tuffs, of Jordan Tuff, zeolitic... phillipsite-rich, possible industrial application of, of Aritain, Jordan CAS REGISTRY NUMBERS: 12174-18-4 in tuffs, of Jordan 4/5/7 (Item 7 from file: 399) DIALOG(R) File 399:CA SEARCH(R) (c) 2001 AMERICAN CHEMICAL SOCIETY. All rts. reserv.

112162348 CA: 112(18)162348c DISSERTATION

A chemical study of the palagonitic tuffs of the Aritain area of Jordan, with special reference to nature, origin, and industrial potential of the associated zeolite deposits

AUTHOR(S): Dwairi, Ibrahim Moh'd Ali

LOCATION: Univ. Hull, Hull, UK,

DATE: 1987 PAGES: 558 pp. CODEN: DABBBA LANGUAGE: English CITATION: Diss. Abstr. Int. B 1989, 50(6), 2319 AVAIL: Univ. Microfilms Int., Order No. BRDX86284

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CA253003 Mineralogical and Geological Chemistry

IDENTIFIERS: palagonite tuff Aritain Jordan, zeolite deposit palagonite tuff Jordan

DESCRIPTORS:

Zeolites, occurrence...

deposits of, industrial potential of, in palagonitic tuffs, of Aritain, Jordan

Tuff, palagonitic...

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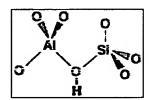
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Chapter 1. Natural Zeolite: Introduction and Properties.

1.1 Introduction

Natural zeolite minerals are secondary minerals and can be defined as crystalline, hydrated alumino-silicates of alkali and alkaline-earth cations that consist of infinite or finite three dimensional crystal structures of (Si, Al)O₄ tetrahedra, which are linked together by the sharing of oxygen atoms (Mumpton, 1983, Flanigen 1983 and Gottardi, 1978), (Figure 1.1). Their structure contains channels and pores filled with a certain amount of water and exchangeable cations. This water can evaporate when heated to about 250°C (dehydration) and is regained at room temperature (re-hydration) (Gottardi, 1985), also some of cations constituent may be exchangeable from the zeolite inner cavities and pores without any major change of zeolite structure (Mumpton, 1983).

Figure. 1.1 Primary building unit of SiO₄ and AlO₄. Tetrahedra



Zeolite minerals were first discovered in Sweden by Cronstedt in 1756, (Gottardi,1978), who gave them their name, which comes from the Greek word meaning the "boiling stones". Since that time, about 50 zeolite natural species have been accounted for, and in the late 1940's, work carried out on developing a synthesis zeolite under hydro-thermal conditions (temperature < 100°C and at normal atmospheric pressure), more than 100 species have been synthesised in the laboratory, which have no natural counterparts (Mumpton, 1978).

1.2 Zeolite Structures

Zeolite structure contains two types of building units namely, primary and secondary. A primary building unit (PBU) is the simpler and is illustrated in figure 1.1, a tetrahedron of (TO₄) of 4 oxygen ions surrounding a central ion of either Si⁴⁺ or Al³⁺. These PBU are linked together to form a three-dimensional framework and nearly all oxygen ions are shared by two tetrahedra (Flangin, 1983; Gottardi,1985). This arrangement reduces the oxygen: silicon ratio to 2:1, and if tetrahedra were centred by Si the chemical formula of its framework would be

Si_nO_{2n}, and the structure would be electrically neutral like Quartz (SiO₂). However, in zeolite structure some of the quadrivalent Si is replaced by trivalent; thus (Al_mSi_{n-m}O_{2n})^m, giving rise to a deficiency of positive charge in the zeolite frameworks, and this is balanced by monoand divalent cations, such as Na⁺, K⁺, Ca²⁺ and Mg²⁺, located outside the tetrahedra; in the channels and pores (Mumpton, 1983, Gottardi, 1985; 1978).

The general formula for natural zeolite according to Gottardi can be given as:

 $M_xD_y [Al_{(x+2y)}Si_{n-(x+2y)}O_{2n}].mH_2O.$

where: $Al_{(x+2y)}Si_{n-(x+2y)}O_{2n}$ represent the framework atom

M: Na⁺, K⁺, or other monovalent cations, and

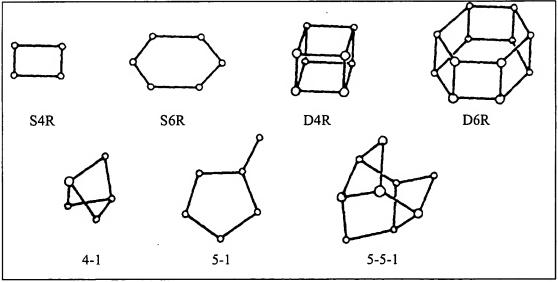
D: Mg²⁺, Ca²⁺, Sr²⁺, Ba²⁺ and other divalent cations, (usually m<=n).

Zeolite structure also contains secondary building units (SBUs), which are formed by the linking of primary building tetrahedral (PBU). They consist of single and double rings of tetrahedra, forming the three dimensional structure of the zeolite material.

Secondary building units may be assembled in different ways to produce different types of frameworks (Figure 1.2). According to Gottardi (1978) the main secondary building units (SBUs) are:

- a.- The 4 ring silicate, single or double (S4R and D4R),
- b.- The 6 ring silicate, single or double (S6R and D6R),
- c.- The fibrous-zeolite unit (4-1),
- d.- The Mordenite-unit (5-1),
- e.- and the stilbite-unit (4-4-1).

Figure 1.2. The main secondary building units (SBUs) of zeolite. (after Gottardi, 1978)



1.3 Formation and Occurrence of zeolite minerals

The formation of sedimentary zeolites can occur by the reaction of volcanic glass (ash) or other alumino-silicate materials with pervading pore waters (ground-, lake-, or seawater). Zeolites are most readily found in alkaline environments (pH>8) because silica is more soluble under these conditions and thus the supply of most essential reactant is greater. Furthermore, because Ca, K, and Na are essential for zeolite structure formations; zeolites tend to form in an environment where these ions are abundant (Hawkins, 1984).

The formation of zeolites in nature is influenced by numerous factors, such as temperature, pressure, reaction time and the activities of dissolved species such as H⁺, silica, alumina, alkaline and earth- alkaline ions.

Natural zeolite deposits are abundant world wide and available in mineable amounts. Their occurrences are mostly in sedimentary rocks and can be categorised into several types of geological environments including: saline – alkaline lakes; saline, alkaline soil systems, deep sea sediments; hydro-thermal alteration systems; hydro-thermal alteration deposits; and burial diagenetic or low-grade metamorphic rocks, (Hawkins,1984; Mumpton 1978).

1.4 Properties of natural zeolite

Zeolite mineral specie have unique properties which are dependent upon its various crystal structures and thus the type of inner cavities; pores; their size and form. Many of these properties are especially desirable for environmental protection, such as cation exchange capacity, ammonium capacity, acid stability, adsorption properties and wet attrition resistance.

1.4.1 Cation-Exchange Capacity (CEC)

Total CEC is one of the most important characteristics that gives zeolite species its importance in environmental protection at an industrial level. Cation exchange capacity is a measure of the number of counter ions present per unit weight or volume of the zeolite and represents the number of cations available for exchange (Semmens, 1984), in other words, it is a function of the degree of Al substitution for Si in the zeolite framework structure; the greater the substitution, the greater the deficiency of positive charge and the greater the number of alkali or earth alkaline cations are required for electrical neutrality, (Table 1.1), (Mumpton, 1984).

Factors which may reduce the exchange capacity;

- 1. The size of zeolite pores may be smaller than the ionic radius of some elements; which leads to cations being completely or partially excluded from exchange; or when the ionic radius of the exchangeable cation is larger than the zeolites pore-volume and / or interconnecting channels and thus leads to ion sieving process (Semmens 1984).
- 2. Cations could be trapped in structural positions (sodalite units) and, therefore, will not be more exchangeable, (Mumpton, 1984).

Table 1.1 The relationship between Si/Al ratio and cation exchange capacity of some natural zeolites; after Colella (1996), * Data calculated from unit-cell formula; (after Mumpton (1984).

Zeolite	Structure	CEC*	Si/Al ratio
	Type cod	Meq/g	ranges
Chabazite	СНА.	3,84	1,43-4,18
Clinoptilolite	HEU.	2,16	2,92-5,04
Erionite	ERI.	3,12	3,05-3,99
Ferrierite	FER.	2,33	3,79-6,14
Heulandite	HEU.	2,91	2,85-4,31
Laumontite	LAU.	4,25	1,95-2,25
Mordenite	MOR.	2,29	4,19-5,79
Phillipsite	PHI.	3,31	1,45-2,87
Faujasite	FAU.	3,39	-

In general, the total cation exchange capacity depends on the type and volume of adsorption sites in zeolite; exchangeable cation sorts; ion radius and charge of cations in the solution, (Semmens and Seyfarth, (1978).

1.4.2 Adsorption Property

The inner structure of zeolite mineral which forms cavities and channels are generally filled with water molecules that form a hydration sphere around the exchangeable cations (such as Ca, Na, K and Mg) (Mumpton, 1984). Much of the water molecules can be removed from the cavities and channels after the zeolite minerals have been heated for several hours at different temperatures between 200 and 350°C; (zeolite dehydration or activation). This permits molecules with a fit diameter to enter the cavities and channels (e.g. water from

atmosphere humidity resulted in zeolite rehydration). Otherwise, a molecule with a large diameter would be excluded (Molecular sieving property). Thus, zeolite minerals have the ability to separate different gases on the basis of its size (Mumpton 1984). Also, polar gases are more preferable to be adsorbed than a non polar molecule (Flanigen 1984); for example CO_2 is more preferable than CH_4 .

There are many factors which contribute to variations of zeolite adsorption properties, such as Si/Al ratio in the zeolite structure, pore volume and size, type of adsorption sites, size and shape of cages and channels in zeolite structure (Flanigen 1984). Natural zeolites (e.g. Clinoptilolite) have many commercial applications because of their adsorption and ion exchange characteristics, these include: purification of acid natural gas streams, drying and separation of air to produce oxygen and nitrogen (Flanigen 1984). Furthermore, the capability of zeolite to capture and immobilise ammonia in its structure makes it important in reducing odour intensities (Ronald Miner 1984).

1.4.3 Extensive Properties

Natural zeolite deposits are mainly soft, friable, and have a small attrition resistance; depending on its formation in the nature. For their economical uses, zeolite deposits should be rich in zeolite minerals of interest. In cases of use as cation exchange and adsorption materials, it should also have a high porosity in order to allow gases and liquids to be diffused between the grains, an acceptable packed bed density which is an important parameter by large scale applications, and the deposits should be soft enough to be crushed to their desired particle size (Mumpton, 1984). Other characteristics of zeolite deposits should also be determined, such as thermal stability and their resistance in acidic solutions.

1.5 Uses of Natural Zeolite

Based on their unique properties, including its low cost, world-wide distribution of zeolite deposits, more than 300.000 tons of zeolitic tuff is used yearly in the United States; Italy; Hungary; Bulgaria and in other countries of the world. Natural zeolites have been utilised in numerous areas of applications, such as ion exchangers in wastewater treatment (domestically-, industrial-, and agricultural origins); as lightweight aggregate in fertilisers and soil conditioners; in pozzolanic cements and concrete; as filler material in paper industry; as dietary supplements in animal husbandry; separation of nitrogen from air; as reforming petroleum catalysis; and other uses (Mumpton, 1978).

1.6 Jordanian Natural Zeolite

In Northeast Jordan in the Aritain area (120km NE Amman city- Appendix 1.1), Zeolite bearings tuff deposits were first discovered by Dwairi in 1984, who showed its presence (mainly containing Phillipsite mineral) in mineable quantities with traces of Chabazite and Faujasite, and suggested the economical utilisation of these Phillipsitic tuffs in industrial applications (Dwairi,1987, 1991). According to his study, zeolitic tuff deposits could be subdivided into three types depending on their degree of zeolitisation as follows:

- a- Least zeolitized tuff (Violet zeolitic tuff).
- b- Moderately zeolitic tuff (Brownish zeolitic tuff), and
- c- Highly zeolitic tuff (Reddish zeolitic tuff).

Furthermore, the formation sequence of Jordanian Phillipsite, in the area of discovery, is explained as a reaction process of basaltic glass with alkaline water (Dwairi, 1987) in the following steps:

- a- The reaction between volcanic glass and pore alkaline water leads to palagonite with a thin film of inter granular phillipsite.
- b- Palagonite react with Mg-rich pore solutions leads to Mg-clay.
- c- Mg-clay by alteration guides to alumino-silicate gel.
- d- By the reaction of this gel with Na⁺- and K⁺- rich pore water, phillipsite mineral will be formed in-situ.

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Zeolites Statistics and Information

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TITLE: A functional tourmaline fertilizer for removing residual agricultural chemical from soil, activating water, improving acidic structure of ground, increasing soil fertility and promoting plant growth

INVENTOR: LI, X; YANG, W; YANG, Z

PATENT-ASSIGNEE: LI X (LIXXI)

PRIORITY-DATA: 2001CN-0113963 (May 18, 2001)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES MA

MAIN-IPC

CN 1320581 A

November 7, 2001

001

C05D009/00

APPLICATION-DATA:

PUB-NO

APPL-DATE

APPL-NO

DESCRIPTOR

CN 1320581A

May 18, 2001

2001CN-0113963

INT-CL (IPC): C05 D 9/00; C05 D 11/00

ABSTRACTED-PUB-NO: CN 1320581A

BASIC-ABSTRACT:

NOVELTY - A functional tourmaline fertilizer for removing residual agricultural chemical from soil, activating water, improving acidic structure of ground, increasing soil fertility and promoting plant growth is prepared from tourmaline (10-50%), Chinese medical stone (25-60%), zeolite (10-50%), dolemite (5-10%), potash feldspar (5-10%), and calcite (5-10%) through crushing, mixing and firing.

ACTIVITY - Fertilizer.

No biological data given.

MECHANISM OF ACTION - None given.

ABSTRACTED-PUB-NO: CN 1320581A

EQUIVALENT-ABSTRACTS:

CHOSEN-DRAWING: Dwg.0/0

DERWENT-CLASS: C04

CPI-CODES: C04-A10; C05-A01A; C14-T;